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REG. NO. 2-9145-2

CONTRACT REPORT NO. OSQ82-00183

March 15, 1983

Dr. C.W. Smith
CANMET
Mineral Sciences Laboratories
Dept. of Energy, Mines & Resources
555 Booth St.
Room 104
Ottawa, Ont.
K1A 0G1

Dear Dr. Smith:

In accordance with my letter of January 11, 1983 to Dr. R.G. Agarwal, the work on project no. 15SQ.23440-2-9145, Radiochemical Determinations for Tailings Reference Materials, has now been completed. The results are listed in Table 1 and the methodologies are described in Appendices I-III for your consideration.

During the course of analyses, we discovered that the materials provided to us were considerably coarser than indicated on the bottles. A screen analysis on a few samples has shown the presence of some +100 mesh material. In addition, approximately 60% of the material is +200 mesh. The CANMET samples UTS-1 and UTS-3 are the most coarse.

Since the analyses for Th-232 as reported in Table 1 were performed at Dr. E.L. Hoffman's laboratory, Nuclear Activation Services, McMaster University, Hamilton, and Th-230 determination was done at Chemex Labs Ltd., Vancouver, it was felt necessary to test the validity of the method used at Chemex Labs. In Table 2 are listed the concentrations of Th-232 determined by both alpha and gamma spectroscopy. The Th-232 activity by alpha spectroscopy is of course measured simultaneously with the Th-230 activity and is used to demonstrate the validity of the Th-230 method. The uncertainties relating to the Th-230 analyses (Table 1) are somewhat higher than requested. This is due to the fact that very small aliquots were used in order to obtain a good alpha spectra, especially for those samples containing Th-232, Th-230, and Th-228.

Please note that the work at Chemex Labs Ltd. was performed under the

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direction of Dr. Michael LeGeyt and the analyses done at Nuclear Activation Services at McMaster University were performed under the guidance of Dr. E.L. Hoffman.

The reference dates for the determination of Pb-210 and Po-210 are listed in Table 3, however, all determinations at Dr. Hoffman's labaratory were done within 12 hours. At the time of determination of Pb-210, Po-210, and Th-230, the following reference standards were used and concentrations of various radioisotopes, measured simultaneously with the test samples, are given below:

Standard Reference	Pb-210 (Bq/g)		Po-210 (Bq/g)		Th-230 (Bq/g)	
CANMET DH-1	23.00	0.40	23.60	0.30		
CANMET DL-2	1.41	0.11	1.37	0.07	1.44	0.07
CANMET BL-1	2.81	0.15			2.88	0.15

I trust that this information is sufficient for your purposes. If you have any questions, please do not hesitate to contact me. Meanwhile, please find enclosed an invoice for the work performed to date.

Yours truly,

MATERIALS RESEARCH LABORATORY LTD.

Sudesh K. Singh, Ph.D.
President

SKS/ad
Encl.

TABLE 1

RADIOISOTOPE CONCENTRATION OF REFERENCE URANIUM TAILINGS

Sample no.	Subsample no.	Radioisotope							
		Pb-210 in Bq/g	Po-210 in Bq/g	Ra-226 in PG/g	Ra-228 in PG/g	Th-228 in PG/g	Th-230 in Bq/g	Th-232 in ppm	U(total) in ppm
UTS-1	1	3.52 ± 0.22	3.44 ± 0.15	20	< 0.10	0.02	2.37 ± 0.15	127	50.8
	2	3.44 ± 0.22	3.22 ± 0.15	30	< 0.10	0.02	2.74 ± 0.15	143	48.3
	3	3.48 ± 0.22	3.26 ± 0.15	40	< 0.10	0.02	2.59 ± 0.15	136	53.0
	4	3.52 ± 0.22	3.03 ± 0.15	10	< 0.10	0.02	2.48 ± 0.15	137	59.0
UTS-2	1	5.33 ± 0.26	4.92 ± 0.22	50	< 0.10	0.02	3.26 ± 0.22	171	58.4
	2	4.85 ± 0.26	4.81 ± 0.22	80	< 0.10	0.02	3.22 ± 0.22	162	58.1
	3	4.70 ± 0.26	5.18 ± 0.22	80	< 0.10	0.02	3.44 ± 0.22	168	57.2
	4	4.60 ± 0.26	4.14 ± 0.22	60	< 0.10	0.02	3.33 ± 0.22	168	54.6
UTS-3	1	13.10 ± 0.40	13.20 ± 0.30	40			10.20 ± 0.60		543.0
	2	13.90 ± 0.40	13.60 ± 0.30	40			10.00 ± 0.60		555.0
	3	14.10 ± 0.40	13.70 ± 0.30	40			10.50 ± 0.60		552.0
	4	13.30 ± 0.40	12.70 ± 0.30	30			11.00 ± 0.70		559.0
UTS-4	1	36.70 ± 0.60	34.80 ± 0.40	190			23.40 ± 1.50		1035.0
	2	35.30 ± 0.60	34.60 ± 0.40	250			22.50 ± 1.40		1041.0
	3	36.60 ± 0.60	34.50 ± 0.40	270			22.80 ± 1.40		1047.0
	4	37.00 ± 0.60	38.00 ± 0.50	230			23.20 ± 1.40		1024.0
UTS-5	1	16.40 ± 0.40	15.70 ± 0.30	70	0.10	0.06	15.90 ± 0.90	467	1294.0
	2	17.00 ± 0.40	17.70 ± 0.30	60	0.20	0.06	16.50 ± 0.90	467	1269.0
	3	17.30 ± 0.40	16.60 ± 0.30	50	0.10	0.06	16.40 ± 0.90	464	1276.0
	4	16.90 ± 0.40	17.70 ± 0.30	30	0.10	0.06	16.40 ± 0.90	459	1305.0

TABLE 2

COMPARISON OF Th-232 CONCENTRATIONS
BY ALPHA AND GAMMA SPECTROSCOPY

Sample no.	Th-232 in ppm	
	Alpha spectroscopy	Gamma spectroscopy
UTS-2	126	141
	145	141
UTS-3	487	480
	469	465
UTS-5	187	170
	171	171
Standard reference CANMET DL-2	80	76

TABLE 3

REFERENCE DATES (1983)

Sample no.	Subsample no.	Pb-210 analysis	Po-210 analysis
UTS-1	1	Feb. 15	Jan. 31
	2	Feb. 15	Jan. 31
	3	Feb. 15	Jan. 31
	4	Feb. 15	Jan. 31
UTS-2	1	Feb. 15	Jan. 31
	2	Feb. 9	Jan. 31
	3	Feb. 9	Jan. 31
	4	Feb. 9	Jan. 31
UTS-3	1	Feb. 3	Jan. 28
	2	Feb. 3	Jan. 28
	3	Feb. 4	Jan. 28
	4	Feb. 4	Jan. 28
UTS-4	1	Feb. 3	Jan. 28
	2	Feb. 14	Jan. 28
	3	Feb. 9	Feb. 15
	4	Feb. 9	Feb. 15
UTS-5	1	Feb. 10	Jan. 27
	2	Feb. 10	Jan. 27
	3	Feb. 10	Jan. 27
	4	Feb. 10	Jan. 27

APPENDIX I

Chemical Procedures Adopted for Determination of Th-228, Th-232, Ra-226, and Ra-228

A. Decomposition

Samples were digested with 5 ml 1:1 H_2SO_4
5 ml concentrated HCl
5 ml concentrated HNO_3
25 ml concentrated HF

taken to dryness. The residue was redissolved in 20 ml of 10% HCl and 10% HNO_3. Any residue was redigested with 10 ml HF and mixed acids, taken to dryness, and redissolved with HCl and HNO_3. These solutions were combined and filtered. The residue was fused with 0.5 g $LiBO_2$ and dissolved in 5% HNO_3.

B. Separation

Ra-226, Ra-228

An aliquot of the digestion solution was taken, diluted, and the pH adjusted to ~8-9. To this solution, lead nitrate and an active barium carrier were added. By adjusting the pH to ~1 with H_2SO_4, the precipitation of $BaRaPbSO_4$ was initiated. This precipitate was filtered and redissolved in EDTA. From the EDTA solution, $BaRaSO_4$ was precipitated and collected on a millipore filter. This was suitably mounted for alpha spectroscopy to determine the Ra-226 content. Yields were determined by gamma counting of the barium carrier.

The above procedure was repeated without the barium carrier (which is itself a β emitter) and sources prepared for β counting to determine the Ra-228 content.

Th-228

An aliquot of the digestion solution was added to an anion exchange column preconditioned with 9N HCl. The column is eluted with 9N HCl to recover the thorium. The thorium fraction is taken slowly to dryness, then refluxed in an HNO_3/HCl mixture, and again taken to dryness. The residue is dissolved in 8N HNO_3 and added to an anion exchange column preconditioned with 0.1N HCl, followed by 8N HNO_3. The thorium is eluted from the column with 0.1N HCl. The eluant is taken to dryness and redissolved in 0.1N HNO_3. The thorium is extracted into 0.5M TTA (2-Thenoyltrifluoroacetone in benzene), and the TTA gently evaporated to reduce the volume. This is transferred dropwise onto a

clean heated stainless steel planchet, and evaporated to dryness in a series of concentric rings. The yield is determined by monitoring the Th-232 content. The Th-228 is determined by α spectroscopy.

C. Reference Standards

Ra-226	NBS Reference Standard Material 4964-B ($101.10 \times 10^{-6} \pm 0.50\%$ g Ra-226)
Ra-228 Th-228 Th-232	Standard Monazite Sand ($1.404 \pm 0.017\%$ Th)

All isolations and counting periods were within 12 hours of each other.

APPENDIX II

Radiochemical Procedures Adopted for Determination of Pb-210, Po-210, and Th-230

The radionuclides Po-210 and Pb-210 were measured by their alpha and beta activity respectively following a radiochemical separation. Their activities were measured using a Canberra Model 2200 Alpha, Beta Analyzer. This instrument consists of an external proportional counter with an ultra-thin detector window. The counter is completely surrounded by four inches of virgin lead and utilizes ultrahigh purity P-10 counting gas (90% argon-10% methane).

A. Pb-210

Because of the weak beta emission from Pb-210, the determination of Pb-210 was carried out indirectly by measurement of its high energy beta-emitting daughter Bi-210.

A mixed carrier solution containing stable lead and bismuth was added to a 0.5 g sample and then digested to dryness with a mixture of nitric, hydrofluoric, and perchloric acids. If any residue remained, the digestion was repeated to yield a clear digest when taken up in 2M HCl. Lead and bismuth were extracted into chloroform with diethyldithiocarbamate and then back extracted into aqueous solution. The bismuth concentration of this aqueous solution was then determined by atomic absorption spectrophotometry to determine the recovery of bismuth (typically 95-100%) through the extraction procedure. The bismuth was subsequently precipitated and purified as the oxychloride. The precipitate was filtered through a 0.45u membrane filter in a ring and disc filter assembly, air-dried briefly, and then covered with aluminum foil. The foil serves to screen out weak beta emission from Pb-210 as well as alpha emission from Po-210 which is carried through the procedure.

Samples were counted for 100 minutes and compared to the activities of standard Pb-210 and Bi-210 solutions carried through the same procedure. Certified reference materials were also analyzed with each batch. The standard Pb-210 solution, with which Bi-210 is certified to be in equilibrium, was purchased from Amersham Radiochemical Ltd. and was certified to have a total uncertainty of not greater than 2.3%.

Ref: Canmet Report No. 78-22, G. Smithson, Ed. (1979) pp. 19-22.

B. Po-210

Samples (0.5 g) for polonium analysis were digested in Teflon beakers with a mixture of nitric, hydrofluoric, and perchloric acids. The mixture was taken to perchloric acid fumes but not to dryness. The digested solutions were diluted, reduced with ascorbic acid then heated for 2 hours in contact with a silver disc. Under these conditions, polonium spontaneously deposits onto the silver disc which is subsequently counted for its alpha activity. To determine the yield of Po-210, samples were analyzed in duplicate, the second set being spiked with standard amounts of Po-210. Yields varied from 86-93%. Samples were counted for 100 minutes and compared to the activity of standard Po-210 solutions carried through the same procedure. Certified reference materials were also analyzed with each batch. The standard Po-210 solution was purchased from Amersham Chemicals and was certified to have a total uncertainty not exceeding 2.3%.

Ref: Canmet Report No. 78-22, G. Smithson, Ed. (1979) pp. 23-28.

C. Th-230

Th-230 was determined by alpha spectroscopy using an Ortec Model 496 alpha spectrometer with two ruggedized silicon surface barrier detectors. The alpha spectrometer has a spectrum storage capability of 120 channels over a 3MeV range. The multichannel analyzer is controlled directly by a PDP 11/34 computer with 256K bytes of memory, 4 hard surface discs, a 9-track 1600 bpi tape drive, and 5 different video or typewriter terminals.

Samples (0.25-0.50 g) for Th-230 analysis were spiked with beta-emitting Th-234 as a radiotracer, lanthanum chloride as a carrier, and barium nitrate as a holdback carrier for radium. This mixture was then digested with nitric, hydrofluoric, and perchloric acids until there was no undissolved residue. Thorium isotopes were then coprecipitated with lanthanum hydroxide, filtered, redissolved, and coprecipitated with lanthanum fluoride. The thorium isotopes were then collected on 0.45u filters and alpha-counted using the instrumentation described above. The samples were also beta-counted for Th-234 to determine the overall yield of the separation. In addition, 2 samples of each replicate group were spiked with Th-230 to verify that counting efficiencies were not compromised by matrix self-absorption.

The activity of samples was compared with the activity of standard Th-230 solutions carried through the same procedure. Certified reference materials

were also analyzed with each batch of samples. The standard Th-230 solution was obtained from Isotope Products Laboratories and had a total uncertainty not exceeding 5.95% in its calibration.

As an additional check, several samples with elevated Th-232 levels were analyzed by neutron activation analysis. The concentration of Th-232 by this method was compared with the activity of Th-232 as determined by alpha spectroscopy. Agreement between the two techniques was good, confirming the validity of the digestion, separation, and counting techniques.

Report on the
Radiochemical Determinations for Tailings
Reference Materials

D.S.S. File No.: 15SQ.23440-2-9145-2

Contract Serial No.: OSQ82-00183

Introduction

This report contains the necessary information for completion of Project No. 23440-2-9145-2 as outlined in section 7, Appendix A of contract serial number OSQ82-00183.

The results contained in this report were determined by the Analytical Services Group, Research and Development Division, Eldorado Resources Limited, located in Tunney's Pasture, Ottawa, Ontario. The project supervisor was Mr. M.C. Miedema. Results for ^{226}Ra were determined by Mrs. G.A. Dunlop. Results for ^{230}Th and ^{210}Pb were determined by Mr. R. Jones and Mr. G.B. Moodie. Results for ^{210}Po were determined by Mr. G.B. Moodie.

Methodology

Sub-samples of 1-2 g (see tables for individual weights) of material were used for all determinations. Samples were solubilized using a mixed flux[1] for ^{226}Ra determination, KF/pyrosulphate fusion[2] for ^{230}Th and ^{210}Pb determinations, and a four acid treatment (HF, HNO_3, H_2SO_4, HCl) for ^{210}Po determinations. After dissolution each sample was carried through one of the following determinations.

Radium-226

Radium-226 was determined exclusively by the ^{222}Rn de-emanation procedure:

Radium-226 (t1/2 = 1622 yr) decays by alpha emission to form radon-222 (t1/2 = 3.8d) gas which in turn produces alpha emitting daughter isotopes.

The method is based on measuring the alpha radiation of radon-222 and its daughters. The radon gas is separated from radium-226 by de-emanation into a cell and counted by a scintillation technique.

The radium was co-precipitated with 100 mg barium as $Ba(Ra)SO_4$. The precipitate was filtered, dissolved, then transferred to a specially designed bubbler and allowed to age (usually for 6 days). Radon-222 gas produced by decay of radium-226 was transferred by aeration with nitrogen gas into a scintillation cell. The cell was allowed to stand for four hours in order for radon-222 to reach equilibrium with its alpha emitting daughter isotopes. The cell was counted on a photomultiplier tube/scaler until > 10,000 counts were recorded. The amount of radium-226 was calculated from the observed counting rate and the period of ingrowth for the radon-222.

Thorium-230

The method used to determine ^{230}Th was as follows:

A sample aliquot was spiked with ^{234}Th tracer and the total thorium precipitated as Pb(Ba, Ra, Th) SO_4 by the addition of $Pb(NO_3)_2$, $BaCl_2$ and $Fe_2(SO_4)_3$. The precipitate was dissolved in

alkaline DTPA. The pH was lowered in the presence of excess sulphate to selectively precipitate radium as $Ba(Ra)SO_4$. The precipitate was filtered off and the thorium precipitated on hydrous titanium dioxide by the addition of Ti^{4+} ion and by raising the pH of the filtrate to 14 with NaOH. This precipitate was filtered on a membrane filter, dried and counted. Beta counting was used to determine chemical recovery of thorium. Alpha counting was used to determine the ^{230}Th activity.

Lead-210

The determination of ^{210}Pb was carried out as follows:

Lead and bismuth carriers were added to the aqueous sample which was 2N in HCl. Ascorbic acid was added to reduce interferring ferric ions. Bismuth along with ^{210}Bi was extracted into diethylammonium diethyldithiocarbamate (DDTC) in chloroform. This organic extract was discarded and ^{210}Bi was allowed to grow back into the aqueous phase for a known period of time. Bi carrier was again added to the aqueous phase and extracted into DDTC/chloroform. The organic phase was wet ashed with nitric acid. The bismuth was taken up in dilute nitric acid and then precipitated as bismuth hydroxide by the addition of ammonia. The bismuth was purified by dissolving the precipitate in concentrated HCl. Bismuth oxychloride was precipitated by dilution with water.

The recovery of Bi through the extraction stage was determined by atomic absorption spectrophotometry. The ^{210}Bi was counted on a low background beta counter. The ^{210}Pb concentration was back-calculated using radiochemical formulas.

Polonium-210

Polonium-210 was determined by its spontaneous deposition on a nickel disc from a hydrochloric acid medium. The disc was then alpha counted. Chemical recoveries were determined by using a ^{208}Po tracer.

Calibration Reference Standards

Table 1 is a listing of standards used both to calibrate the various detectors and as controls during the actual analyses. All standards used for calibration purposes were reported by their manufacturer as being traceable to NBS standards in assigning values.

Results

Tables 2-6 contain the results for all determinations carried out. Sub-sample sizes for each determination are also reported. A larger than expected variation in the results for ^{226}Ra and ^{210}Pb was noted for sample UTS-2. Our laboratory carried out 11 determinations for each of these two radionuclides instead of the required four. These have been reported separately in Table 6. No reason for this variability was determined. These extra determinations are provided free of charge.

Table 1. Calibration and control standards.

Isotope	Source	Uncertainty % (99.7% C.L)	Use
^{226}Ra	Amersham RA31	< 5	Calibration/ Control
^{226}Ra	U.S.E.P.A. 1907-1-39	10.2	Control
^{230}Th	U.S.E.P.A. 1838-3-38	5.7	Calibration/ Control
^{210}Pb	Amersham RBZ.44	1.9	Calibration/ Control
^{210}Po	Amersham PDZ.44	3.4	Control
$^{230}Th/^{226}Ra$ $^{210}Pb/^{210}Po$	U.S.E.P.A. Standard Pitchblende Ore	3.2	Control
$^{230}Th/^{226}Ra$ $^{210}Pb/^{210}Po$	U.S.E.P.A. Diluted Pitchblende Ore	17	Control

Table 2. ^{230}Th results.

Sample	Sub-Sample Mass g	Result Bq/g
UTS-1	1.0065	3.01±0.06
UTS-1	1.0138	3.15±0.06
UTS-1	1.0182	3.21±0.06
UTS-1	1.0514	3.02±0.06
UTS-2	1.0398	3.06±0.06
UTS-2	1.0375	3.33±0.06
UTS-2	1.0175	3.18±0.06
UTS-2	1.0193	3.14±0.06
UTS-3	1.0100	10.9±0.2
UTS-3	1.0067	10.6±0.2
UTS-3	1.0125	10.4±0.2
UTS-3	1.0039	10.4±0.2
UTS-4	2.0004	24.4±0.4
UTS-4	2.0014	24.6±0.4
UTS-4	2.0253	22.6±0.4
UTS-4	1.0820	23.0±0.4
UTS-5	2.0284	15.3±0.3
UTS-5	1.6294	15.7±0.3
UTS-5	2.0331	14.3±0.3
UTS-5	1.1694	15.8±0.3

Table 3. ^{226}Ra results.

Sample	Sub-Sample Mass g	Result Bq/g
UTS-1	1.0541	3.38±0.03
UTS-1	1.0425	3.65±0.04
UTS-1	2.0066	3.44±0.03
UTS-1	1.6365	3.33±0.03
UTS-2	*	*
UTS-2	*	*
UTS-2	*	*
UTS-2	*	*
UTS-3	2.0288	13.12±0.08
UTS-3	1.1130	12.01±0.09
UTS-3	1.6483	12.81±0.10
UTS-3	1.0019	12.85±0.10
UTS-4	2.0004	39.4±0.2
UTS-4	2.0014	36.5±0.3
UTS-4	2.0253	35.0±0.3
UTS-4	1.4422	39.5±0.3
UTS-5	2.0284	16.20±0.09
UTS-5	1.6294	14.76±0.12
UTS-5	1.1746	16.09±0.10
UTS-5	2.0387	15.04±0.11

*Reported in seperate table

Table 4. ^{210}Pb results.

Sample	Sub-Sample Mass g	Result Bq/g
UTS-1	2.0066	3.27±0.04
UTS-1	1.6733	3.24±0.04
UTS-1	1.2616	3.23±0.04
UTS-1	1.2728	3.15±0.04
UTS-2	*	*
UTS-2	*	*
UTS-2	*	*
UTS-2	*	*
UTS-3	1.0100	12.0±0.2
UTS-3	1.0067	12.7±0.2
UTS-3	1.0512	11.9±0.2
UTS-3	1.0039	12.7±0.2
UTS-4	2.0004	35.9±0.4
UTS-4	2.0014	35.2±0.4
UTS-4	2.0253	35.9±0.4
UTS-4	1.0719	35.6±0.4
UTS-5	2.0284	14.3±0.2
UTS-5	1.6294	14.8±0.2
UTS-5	1.2656	14.7±0.2
UTS-5	1.2198	14.7±0.2

*Reported in seperate table

Table 5. ^{210}Po results.

Sample	Sub-Sample Mass g	Result Bq/g	Date of Analysis
UTS-1	1.1246	3.00±0.05	83/1/24
UTS-1	1.1497	2.81±0.05	83/1/25
UTS-1	1.2616	3.03±0.05	83/1/26
UTS-1	1.2728	2.85±0.05	83/1/27
UTS-2	1.1082	4.26±0.07	83/1/24
UTS-2	1.2015	4.33±0.07	83/1/25
UTS-2	1.0759	4.66±0.07	83/1/26
UTS-2	1.1544	4.36±0.07	83/1/27
UTS-3	1.1273	11.11±0.12	83/1/28
UTS-3	1.0856	9.99±0.12	83/1/25
UTS-3	1.0512	11.03±0.12	83/1/26
UTS-3	1.0093	10.99±0.12	83/1/27
UTS-4	1.0820	33.6±0.5	83/1/25
UTS-4	1.0545	34.0±0.5	83/1/26
UTS-4	1.1279	33.9±0.5	83/1/26
UTS-4	1.0719	36.4±0.5	83/1/27
UTS-5	1.0854	14.5±0.2	83/1/25
UTS-5	1.2680	15.1±0.2	83/1/26
UTS-5	1.1635	16.2±0.2	83/1/27
UTS-5	1.1694	15.5±0.2	83/1/27

Table 6. ^{226}Ra and ^{210}Pb results for UTS-2.

^{226}Ra		^{210}Pb	
Sub-Sample weight (g)	Result Bq/g	Sub-Sample Weight (g)	Result Bq/g
1.1175	5.01±0.05	2.0009	5.48±0.09
2.0009	6.06±0.06	1.6541	4.19±0.09
1.6541	5.06±0.04	1.6678	3.69±0.09
1.6678	7.01±0.05	1.6438	3.99±0.09
1.6438	4.77±0.05	2.0009	5.17±0.09
1.2656	6.19±0.04	1.6541	4.00±0.09
1.2198	5.68±0.05	1.1082	5.59±0.09
1.8047	3.48±0.03	1.2015	4.84±0.09
1.1281	5.76±0.05	1.1544	4.66±0.09
1.5031	5.13±0.05	1.2656	3.15±0.09
1.5132	5.42±0.05	1.2198	3.97±0.09

References

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2. Sill, C.W., Decomposition of Refactory Silicates in Ultramicro Analysis, Analytical Chemistry 33, (1961) 1684-1686.